
⁽¹²⁾ UK Patent Application ⁽¹⁹⁾ GB ⁽¹¹⁾ 2 016 016 A

(21) Application No 7908128

(22) Date of filing

7 Mar 1979

(23) Claims filed

7 Mar 1979

(30) Priority data

(31) 885833

(32) 13 Mar 1978

(33) United States of America
(US)

(43) Application published

19 Sep 1979

(51) INT CL² C08K 3/22

C08L 31/04

(C08L 31/04 23/28)

(52) Domestic classification

C3K 120 125 129 201

EA

(56) Documents cited

GB 1476456

GB 1400162

US 4014770A

US 4006114A

(58) Field of search

C3K

C3V

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(54) Flame retardant polymeric
materials

(57) A flame retardant composition exhibiting enhanced properties comprises a polymer such as a vinyl acetate-ethylene copolymer, chlorinated polyethylene or other halogenated polymer, and hydrated alumina. No electrical conductor coated with such a polymeric composition is a particularly important application, e.g. in automotive wiring.

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SPECIFICATION

Flame retardant polymeric materials

- 5 This invention relates to polymeric flame retardant compositions. 5
- Industry is continually searching for new fire resistant polymeric compositions to improve the performance of existing products and/or meet the needs of new applications. One of the most important areas where fire resistant polymeric compositions find use is in the electrical environment where physical, insulating and fire resistant properties are sought. A particularly important application in this area is insulation used in automotive vehicle electrical wiring. This type of wiring also requires for many applications resistance to oil. Typical applications include primary wiring, spark plug cables, ignition wiring and battery cables. Another important application is for insulated wire suitable for use as a fusable link in automotive wiring harnesses. Good physical properties are extremely important in this application to minimize insulation rupture due to short circuits which may cause an explosion. 10 15
- Materials presently in use for automotive applications include chlorosulphonated polyethylene, ethylene-propylene elastomer, chlorinated polyethylene, silicon elastomer and flame retardant crosslinked polyolefin. Unfortunately, however, these materials are unsuitable for use as, e.g., a fusable link in automotive vehicle wiring, e.g. harnesses, because they do not provide the combination of flame retardancy, physical strength, resistance to oil, moisture, heat, gasoline and solvents and insulation rupture due to a severe electrical overload. Additionally, it is very important that the material be easily processed, e.g. by extrusion, into its final form. Performance of these materials in automotive electrical insulation applications is described in detail in the product brochures and applications literature distributed by producing companies for these materials. 20 25
- Reference books are replete with discussions of mechanisms of fire retardancy and operative systems. "Flame Retardancy of Polymeric Materials", Volume 1, by W. C. Kuryla and A. J. Papa, Marcel Dekker, Inc., N.Y., 1973, Chapter 1 and pages 171-181 and Lyons, "The Chemistry & Uses of Fire Retardants", John Wiley and Sons, Inc., 1970, pages 330-332, are exemplary. A number of U.S. patents show the wide variety of fire retardant additive combinations known in the art. U.S. Patent No. 3,832,326 discloses crosslinkable polymeric compositions based upon an ethylene-vinyl acetate copolymer, preferably containing less than about 28% vinyl acetate by weight, having improved moisture, heat resistance and flame retardance and is particularly adapted for coating electrical wiring. The compositions are specifically directed to a non-halogenated flame retardant system. Another non-halogenated system is shown in U.S. Patent No. 3,741,929. U.S. Patent No. 3,362,928 shows a thermoset system based on a diallyl phthalate molding compound, a flame retardant organic chlorine containing compound, antimony oxide and hydrated alumina. The compositions are used for the manufacture of rigid type electronic components and switches. U.S. Patent No. 3,694,305 discloses an emulsion type adhesive for laminating various plies of a flame retardant fire barrier. U.S. Patent No. 3,720,643 discloses a fire retardant polymeric material based on adding talc, chlorinated polyethylene and antimony trioxide to polypropylene, styrene-acrylonitrile copolymer or acrylonitrile-butadiene-styrene resins. 30 35 40
- U.S. Patent No. 3,936,403 relates to synthetic resin compositions having flameproofness and surface hardness comprising an olefinic resin, vinyl chloride resins and alumina trihydrate having a gibbsite crystal structure. Ethylene-vinyl acetate copolymers are not disclosed and the preferred resin is high density polyethylene, which, as shown hereinbelow, cannot be extruded to make a coating for an electrical conductor. Further, the disclosed vinyl chloride resin is polyvinyl chloride which, again, cannot be employed in the claimed composition since the coated wire does not pass the UL-FR-1 flame test. 45 50
- It has now been unexpectedly discovered that fire retardant polymeric compositions exhibiting enhanced properties suitable for use as insulation for automotive vehicle wiring, comprise suitable proportions of a vinyl acetate-ethylene copolymer, chlorinated polyethylene and hydrated alumina.
- 55 In general, the polymeric fire retardant composition comprises a vinyl acetate-ethylene copolymer containing about 20% to 90% by weight vinyl acetate and, per hundred parts of the copolymer, about 10 to 50 parts of chlorinated polyethylene and about 70 to 300 parts of hydrated alumina. A preferred composition comprises about 10 to 35 parts of chlorinated polyethylene and about 100 to 250 parts of hydrated alumina. A composition wherein the copolymer contains vinyl acetate in an amount of about 40% to 70% is highly preferred. 60
- Although not necessary to achieve the desired properties, it is advantageous to employ antimony trioxide and/or silica to further improve the fire resistance and processability, respectively, of the composition. In general, the antimony oxide and silica each may be used in an amount, per hundred parts of the copolymer, of about 10 to 50 parts, preferably about 10 to 35 parts. A silane coupling agent as discussed hereinbelow is necessary to provide a 65

composition which may be used as a coating for wire and cable.

Such polymeric fire retardant compositions find particular utility in the insulation of automotive wire but are useful in other applications where a combination of enhanced properties are needed such as good processability, physical properties, electrical properties, oil resistance, moisture resistance, gasoline or solvent resistance and fire resistance.

The polymeric component of the present composition is based upon a vinyl acetate-ethylene copolymer. The copolymer contains vinyl acetate (VA) in an amount, by weight, of about 20% to about 90%, preferably about 40% to 70%, e.g. 50% to 65%. The preferred copolymers have a combination of properties which are mainly dependent on the vinyl acetate content. For example, as the vinyl acetate content is increased, the oil and solvent resistance are generally increased. A more detailed description of the preferred copolymer is set forth in the booklet entitled "Vynathene® VAE Elastomers" by National Distillers and Chemical Corporations. In general, the preferred copolymers have a density of about 0.960 to 1.05 grams per cubic centimeter (g/cc), a melt flow rate at 125°C. of about .1 to 20 g/10 minutes of flow, vinyl acetate content of about 40% to 70% by weight and inherent viscosities of 0.70 to 1.10 for 0.15 g polymer per 100 ml. tetrahydrofuran at 40°C. Three preferred copolymers which are products of National Distillers and Chemical Corporation are:

(a) VYNATHENE EY 904, a vinyl acetate-ethylene copolymer having a VA content of from about 50% to about 54% by weight and a melt flow rate (MFR) of from about 0.5 to about 1.5 at 125°C.;

(b) VYNATHENE EY 905, a vinyl acetate-ethylene copolymer having a VA content of from about 50% to about 54% by weight and an MFR of from about 1.5 to about 7.0 at 125°C.; and

(c) VYNATHENE EY 907, a vinyl acetate-ethylene copolymer having a VA content of from about 58% to about 62% by weight and an MFR of from about 1.0 to about 2.2 at 125°C.

Although not necessary, to provide a special combination of properties minor amounts of other polymers or copolymers or mixtures thereof may be included in the composition of this invention, e.g., polyethylene, polypropylene, ethylene propylene elastomer, polybutylene, ethylene-acrylate copolymer, ethylene-vinyl chloride copolymer and the like. They may be present in amounts, per hundred parts of vinyl acetate-ethylene copolymer, up to about 25 parts or higher, and are preferably below about 15, e.g. 10, parts.

Chlorinated polyethylene (CPE) is a well-known material and preferred materials are CPE X02242.46 and CPE 4814 manufactured by Dow Chemical Co. Other chlorinated polymers include chlorinated polypropylene, polyvinylidene chloride, chlorosulfonated polyethylene and the like. Other halogenated polymers are useful, especially brominated polymers; such polymers may be found in the text by W. C. Kuryla and A. J. Papa, supra. A composition containing polyvinyl chloride has been found to be unacceptable since it does not pass the UL-FR-1 test for coated wire. CPE is preferred because of its demonstrated effectiveness.

The hydrated alumina, or alumina trihydrate, is preferably included in relatively fine particle sizes of about 0.3 to 2 microns, although larger or smaller size particles may be employed.

Preferred materials are HYDRAL 710 and P. G. Alumina manufactured by Aluminum Co. of America.

An important feature of the invention comprises specially correlating the CPE and hydrated alumina with the vinyl acetate-ethylene copolymer as set forth hereinabove to provide a highly preferred composition having an excellent combination of properties, including passing the stringent requirements of flame tests such as UL-FR-1.

It has been found, unexpectedly, that the combination of the halogenated polymer and hydrated alumina may provide a synergistic flame retardancy effect in the polymer composition of the invention. Thus, for example, other known halogenated fire retardant additives do not exhibit such an increase in fire resistance when combined with hydrated alumina and actually decrease the fire resistance of the material. It has been theorized in U.S. Patent No. 3,694,305, supra, that the combination of a chlorinated paraffin, antimony oxide and hydrated alumina provides a faster synergistic fire retardancy effect. While not wishing to be bound by any theories, it is hypothesized that the combination of the halogenated polymer and hydrated alumina provides a synergistic fire retardancy effect by both materials acting in concert to provide a very effective sustained fire retardancy effect. This effect is particularly important under stringent fire retardancy requirements such as the UL-FR-1 test and is achieved under the combinations of the halogenated polymer and hydrated alumina as discussed hereinabove.

While the amounts of halogenated polymer and hydrated alumina may be varied widely as set forth hereinabove, to achieve a still more enhanced combination of properties it is preferred that high amounts of both components not be employed. This, as shown hereinbelow in the Examples, increasing the amount of halogenated polymer at a high hydrated alumina content, depresses the % elongation of the composition. It is important therefore for some applications to correlate the amount of halogenated polymer and hydrated alumina and it is highly preferred that, per hundred parts of resin, when the hydrated alumina is above about 150 parts, the

halogenated polymer be up to about 15 parts.

Antimony trioxide is the preferred antimony compound, although many other antimony compounds are suitable as known in the art, such as antimony sulfide and sodium antimonite and the organic antimony compounds such as antimony butyrate and antimony caprylate.

The silica component may be any of the well-known materials and preferred silicas include Hi-Sil (Registered Trade Mark) 233 and Hi-Sil EP, both of which are manufactured by PPG Industries, Inc. The particle size of the silica is preferably about 0.01 to 0.05 microns, although larger or smaller size particles may be employed.

The polymeric compositions of this invention may include other ingredients, additives and agents, depending upon the intended service of the products thereof, and the required or desired properties. For example, other components may comprise antioxidants, acid acceptors, preservatives, lubricants and processing aids, mold release agents, pigments or coloring agents, inorganic fillers, waterproofing agents, coupling agents, etc.

The preferred antioxidants are the polyquinolines such as polytrimethyl dihydroquinoline which is sold under the trademark Agerite Resin D and Agerite MA (higher molecular weight) by the R. T. Vanderbilt Company, Inc. Other conventional antioxidants, e.g., useful in the prior art for the stabilization of low density polyethylene and ethylene copolymers, may also be employed. The amount of antioxidant used is about 0.25 to 4% by weight of the total composition.

The preferred acid acceptor is tetrabasic lead fumarate which is marketed under the trademark Lectro 78 by N. L. Industries, Inc. Other acid acceptors such as magnesium oxide, litharge and the like may be utilized. The concentration is about 0.5 to 4% by weight of the total composition or about 1 to 3% of the total resin content.

Lubricants and processing aids, such as stearic acid (Hystrene (Registered Trade Mark) 9718 sold by Humko-Sheffield Chemicals Co.) and calcium stearate are preferred. Others well-known in the art may also be employed. Amounts of about 1 to 3 parts, preferably about 1.5 to 2.5 parts per hundred parts of resin are generally employed.

A coupling agent is preferred in the compositions to provide a composition suitable for coating wire or cable and any coupling agent may be employed in the compositions, it being important that it does not interfere with polymer crosslinking or degrade during polymer processing. Silanes as disclosed in U.S. Patent No. 3,832, 326, supra, incorporated herein by reference, are preferred; e.g., Silane A-172 sold by Union Carbide Co. provides excellent results. Amounts of about 0.2 to 4%, preferably 0.2 to 2% and most preferably 0.5 to 1.2% by weight of the composition are generally employed.

An important feature of the invention is to cross-link the above described compositions to provide their final product form. Crosslinking may be accomplished by any of the known crosslinking techniques, such as chemical crosslinking and irradiation.

Curing agents which can be used herein include such peroxides as: t-butyl perbenzoate, dicumyl peroxide, 2,5-dimethyl-2,5-di(t-butyl peroxy) hexane, 2,5-dimethyl-2,5-di(t-butyl peroxy) hexyne-3, 1,3,5-tris [α,α -dimethyl- α -(t-butyl peroxy)]-methyl benzene, α,α' -bis(t-butyl peroxy) diisopropyl benzene and N-butyl-4,4-bis (t-butyl peroxy) valerate. These curing agents can be used alone or in combination with any of several polyfunctional monomers such as triallyl cyanurate, triallyl isocyanurate, triallyl phosphate, trimethylol propane triacrylate, diallyl fumarate, pentaerythritol tetraacrylate, trimethylol propane trimethacrylate, 1,3-butylene glycol dimethacrylate, allyl methacrylate, ethylene glycol dimethacrylate and 1,3-butylene glycol diacrylate. The preferred curing agents for use herein include Vul-Cup 40 KE alone [40% α,α' bis(t-butyl peroxy) diisopropyl benzene on Burgess KE obtained from Hercules Inc.] and Vul-Cup 40 KE in combination with the polyfunctional monomer triallyl isocyanurate (TAIC) obtained from Allied Chemical Corporation.

The amount of peroxide curing agent can range from about 1.0 to about 10 parts, and preferably from about 3.0 parts to about 6.0 parts per hundred parts of copolymer.

The polyfunctional monomer used as auxiliary curing agent in combination with crosslinking peroxide can be used in the range of from about 0.1 to about 3.0 parts per hundred parts of copolymer. The preferred amount of auxiliary curing agent can range from about 0.5 to about 1.5 parts and the most preferred amount about 1.0 part per hundred parts of copolymer.

The compositions of the invention may be prepared using known compounding techniques. Mixing is preferably carried out using an intensive mixer such as the Banbury or Werner & Pfleiderer mixers. A preferred method is to prepare a polyblend of the resin components and the chlorinated polyethylene. Any other ingredient, excluding the crosslinking agents, may then be added and mixed. If silica is employed, it is preferred to add it before the hydrated alumina.

After thorough mixing, the temperature is raised to about 250°F. and mixing continued for about 1 to 2 minutes. The temperature is then lowered to below about 235°F. and the crosslinking agents added. Mixing is continued until the composition is uniform, usually about 4 to 5 minutes. The batch may then be further processed on a two-roll mill or in a processing extruder.

The compositions of the present invention will now be more fully illustrated by the following

specific examples and are not to be considered as constituting a limitation on the present invention. All parts and percentages given are by weight and temperatures in degrees Fahrenheit unless otherwise indicated.

5 EXAMPLE I

The following compositions as shown in Table 1 were prepared as described hereinbelow in a Banbury mixer, Model No. BR.

Composition 1 was prepared by mixing the resin and CPE for about three minutes at 230 revolutions per minute (rpm.) speed. Hi-Sil 233, Agerite and antimony trioxide were added to the Banbury and mixing continued for about three minutes. The temperature was raised to about 250°F. and mixing continued at this temperature for about one to two minutes. The temperature was then lowered below about 235°F. and the stearic acid and Lector 78 were added and mixed for about three minutes, followed by adding the Vul-Cup 40 KE and continued mixing for about four to five minutes. The batch was then milled on a 6" × 12" Thropp 2 roll rubber mill at about 100°F. and sheeted to 10–15 millimeter (mm.) crepe, which was diced to cubes of about 1/8 inch. Wire coated samples were prepared by extrusion onto 20 gauge stranded wire at 30 mil wall thickness using a 3/4 inch Brabender Extruder, Type No. PL-V340. Curing was performed in a vulcanizing tube at 400°F for 6 minutes. The insulated wire was cooled by quenching in cold water for two minutes under pressure and then retained in water during slow removal of pressure over ten minutes time.

Compositions 2, 3 and 4 were prepared by mixing the resin, hydrated alumina, silane and Agerite for about 5 minutes. The temperature was then raised to about 250°F. and the same procedure for composition 1 was then followed except that calcium stearate replaced Lector 78 and was omitted in composition 4.

Compositions 5 and A were prepared by mixing the resin and CPE for about 3 minutes at 230 rpm. speed. Hi-Sil 233, Agerite and the silane were added and mixing continued for about 3 minutes, followed by addition of the alumina and antimony trioxide and continued mixing for about 3 minutes. The temperature was then raised to about 250°F. and the procedure set forth for Composition 1 was followed.

Compositions B and C were prepared by mixing the resin and CPE for about 3 minutes at 230 rpm. speed. Alumina, Agerite and the silane were then added and mixing continued for about 5 minutes. The temperature was then raised to about 250°F. and the procedure set forth for Composition 1 was followed except that calcium stearate replaced the Lector 78.

Table 1

	Compositions								
	1	2	3	4	5	A	B	C	
Vinyl Acetate-Ethylene									
40 Copolymer ^a	100	100	100	100	75	75	90	80	40
CBE ^b	28	—	—	—	25	25	10	20	
P.G.Alumina	—	175	225	100	50	100	225	225	
Calcium Carbonate	—	—	—	75	—	—	—	—	
Hi-Sil 233	20	—	—	—	20	20	—	—	
45 Antimony Trioxide	35	—	—	—	25	25	—	—	45
Silane A-172	—	4	4	1.5	1	2	4	4	
Agerite MA	2	1	1	2	2	2	1	1	
Lector 78	1.4	—	—	—	1.4	1.4	—	—	
Calcium Stearate	—	2	2	—	—	—	2	2	
50 Stearic Acid ^c	2	2	2	2	2	2	2	2	50
Vul-Cup 40 KE	4	4.25	4.25	4.25	4	4	4.25	4.25	

^a Vynathene EY 904

^b Chlorinated polyethylene manufactured by Dow Chemical Co. and sold under the name CPE

55 4814

^c Hystrene 9718

The cured compositions were tested using the following standard techniques and the results are shown hereinbelow in Table 2: Tensile and Elongation – ASTM D 638; Oxygen Index – ASTM D 863; FR-1 Flame Test – UL-FR-1; Shore A Hardness – ASTM D 2240.

60

Table 2

	Compositions								
	1	2	3	4	5	A	B	C	
5 <i>Unaged</i>									5
Tensile, psi	2850	1290	1820	1400	1890	1570	1980	1820	
Elongation, %	590	130	110	310	170	300	420	140	
Oxygen Index									
10 (O.I.)	30.3	33.0	44.6	31.2	52.2	49.0	37.4	58.4	10
FR-1 Flame									
Test	Fail	Fail	Fail	Fail	Fail	Pass	Pass	Pass	
Shore A									
Hardness	-	-	-	-	92	83	74	92	15

Compositions A, B and C were tested in the aged condition (70 hours at 125°C) and the results are shown hereinbelow in Table 3. Volume Swell (%) was performed with ASTM #3 oil as prescribed in SAE J 878a specifications for fusible link insulation and ASTM Method D-471.

Table 3

	Compositions			
	A	B	C	
25 Tensile, psi	1980	2050	2340	25
Elongation, %	220	350	110	
Shore A Hardness	85	79	93	
Volume Swell (%)	77.3	100	58.7	

30 Volume Swell (%) was performed on Compositions 1, 3 and 4 and the results are 166%, 74.4% and 96.8%, respectively.

A review of the results shown in Tables 2 and 3 shows the unexpected and synergistic performance provided by the compositions of the invention. A comparison of 1, 2, 5 and A clearly shows the importance for flame resistance of using chlorinated polyethylene in combination with hydrated alumina and employing the hydrated alumina above about 70 parts per hundred parts of copolymer. Composition A is particularly preferred because of its good overall properties and its ease of processability. A comparison of B and C shows the importance of using correlated amounts of chlorinated polyethylene and hydrated alumina to achieve enhanced 40 % elongation properties.

EXAMPLE II

To further demonstrate the invention, the following comparative compositions were prepared and tested in accordance with the procedures set forth in Example I. Composition A was 45 repeated and is herein termed A'. Composition A' was repeated replacing the vinyl acetate-ethylene copolymer (VAE) with high density polyethylene (HDPE) (Petrothene LB 830 manufactured by National Distillers and Chemical Corp.) and is termed Composition 6. Composition A' was repeated by (1) replacing the CPE with polyvinyl chloride (PVC) (B.F. Goodrich Geon 103 EP) (Composition 7) and (2) replacing the CPE with PVC plasticized with 25% by weight dioctyl 50 phthalate (Composition 8) both at an equivalent chlorine level based on weight. Composition A' was repeated replacing the VAE with an ethylene-vinyl acetate copolymer (Ultrathene UE 630 made by National Distillers and Chemical Corp.) containing about 18% by weight vinyl acetate and is termed Composition 9. The test results in the unaged condition are as follows:

TABLE IV

	Composition	Tensile (psi)	Elongation, %	Oxygen Index (O.I.)	FR-1 Flame Test	
5	A'	1400	360	41.4	Pass	5
	6	2800	10	32.0	*	
	7	1360	160	42.5	*	
	8	1390	220	40.4	Fail	
10	9	1640	60	36.5	Fail	10

* Couldn't extrude onto wire.

As is clearly shown by the test results, Composition 6 containing HDPE and Composition 7 containing PVC could not be extruded onto wire. Composition 8, containing plasticized PVC failed the FR-1 flame test. Composition 9 containing an ethylene-vinyl acetate copolymer with only 18% VA also failed the FR-1 flame test.

Composition A' without the silane component, could not be extruded onto wire and had an O.I. of 34.9.

While the invention has been directed to copolymers of vinyl acetate and ethylene containing greater than about 20% by weight vinyl acetate, it will be understood to those skilled in the art that the disclosed combination of ingredients is applicable to other polymer and/or copolymer systems which provide the same results afforded by the preferred vinyl acetate-ethylene copolymers, and that such other embodiments are, therefore, also embraced within the scope of the present invention.

CLAIMS

1. A polymeric fire retardant composition comprising vinyl acetate-ethylene copolymer containing 20% to 90% by weight vinyl acetate, and, per hundred parts by weight of the said copolymer, 10 to 50 parts chlorinated polyethylene and 70 to 300 parts hydrated alumina.
2. A composition in accordance with claim 1 wherein the said copolymer contains 40% to 70% by weight vinyl acetate.
3. A composition in accordance with claim 1 or 2 containing, per 100 parts by weight of the said copolymer, 10 to 35 parts chlorinated polyethylene and 100 to 250 parts hydrated alumina.
4. A composition in accordance with any of claims 1 to 3 further containing a silane component in an amount, by weight of the composition, of 0.2 to 4%.
5. A composition in accordance with claim 4 containing 0.2 to 2% silane.
6. A composition in accordance with any of claims 1 to 6 further containing, per hundred parts of the said copolymer, 10 to 50 parts antimony trioxide and/or 10 to 50 parts silica.
7. A composition in accordance with any of claims 1 to 6 wherein the composition is crosslinked.
8. An electrical conductor coated with a composition according to any of claims 1 to 7.
9. A process for preparing a polymeric fire retardant composition which comprises blending a mixture of vinyl acetate-ethylene copolymer containing 20% to 90% by weight vinyl acetate, and, per hundred parts by weight of the said copolymer, 10 to 50 parts chlorinated polyethylene, and 70 to 300 parts hydrated alumina.
10. A process according to claim 9 wherein the said copolymer contains 40% to 70% by weight vinyl acetate.
11. A process according to claim 9 or 10 wherein the mixture contains, per 100 parts by weight of the said copolymer 10 to 35 parts of chlorinated polyethylene and 100 to 250 parts of hydrated alumina.
12. A process according to any of claims 9 to 11 wherein the mixture contains a silane component in an amount by weight of the mixture, of 0.2 to 4%.
13. A process according to claim 12 wherein the silane constitutes 0.2 to 2% by weight of the mixture.
14. A process according to any of claims 9 to 13 wherein the mixture contains per hundred parts of the said copolymer, 10 to 50 parts antimony trioxide and/or 10 to 50 parts silica.
15. A process according to any of claims 9 to 14 including the step of coating the composition on an electrical conductor.
16. A process according to any of claims 9 to 15 wherein the mixture contains a crosslinking agent.
17. A process according to any of claims 9 to 16 including the step of crosslinking the composition.
18. A flame retardant polymeric composition substantially as described in Example A, B or C.

19. A process for preparing a flame retardant polymeric composition, the process being substantially as described in Example A, B or C.

20. A polymeric fire retardant composition comprising vinyl acetate-ethylene copolymer containing 20% to 90% by weight vinyl acetate, and, per hundred parts by weight of the said
5 copolymer, 10 to 50 parts halogenated polymer and 70 to 300 parts hydrated alumina.

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Printed for Her Majesty's Stationery Office by Burgess & Son (Abingdon) Ltd.—1979.
Published at The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.